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(54) PRODUCTION OF WATER-ABSORPTIVE RESIN OF EXCELLENT DURABILITY

(57)Abstract:

PURPOSE: To obtain the title resin excellent in the durability and liquid permeability of a swollen gel and showing a high water absorption rate even to physiologic saline by subjecting an aqueous solution containing a water-soluble, ethylenically unsaturated monomer, a crosslinking agent and a water-soluble chain transfer agent each in a specified amount to aqueous solution polymerization.

CONSTITUTION: An aqueous monomer solution of a concentration of from 30wt.% to the saturation concentration containing a water-soluble, ethylenically unsaturated monomer (e.g. acrylic acid/sodium acrylate mixture), 0.005-5mol%, based on this monomer, crosslinking agent (e.g. trimethylolpropane tri-acrylate) and 0.001-1mol%, based on this monomer, water-soluble chain transfer agent (e.g. sodium hypophosphite) is subjected to aqueous solution polymerization.

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Japanese Unexamined Patent Publication No. 179008/1991 (Tokukaihei 3-179008)

A. Relevance of the Above-identified Document

The following is a partial English translation of exemplary portions of non-English language information that may be relevant to the issue of patentability of the claims of the present application.

- B. <u>Translation of the Relevant Passages of the Document</u>

 See also the attached English Abstract.
- 1. Title of the invention
 PRODUCTION METHOD OF WATER-ABSORBING RESIN
 EXCELLENT IN DURABILITY

2. Claims

1. A production method of water-absorbing resin (D) excellent in durability, comprising: performing aqueous polymerization using a monomer (A) aqueous solution that has a concentration in the range of from 30% by weight to a saturation concentration and contains (i) water-soluble ethylene-based unsaturated monomer (A), (ii) 0.005 mol% to 5 mol% of cross-linking agent (B) with respect to monomer (A), and (iii) 0.001 mol% to 1 mol% of water-soluble chain transfer agent (C) with respect to monomer (A).

- 2. A production method of water-absorbing resin (F) excellent in durability, comprising cross-linking in the vicinity of a surface of water-absorbing resin (D) is cross-linked by a hydrophilic cross-linking agent that is functional contained reactive with group water-absorbing resin (D), which water-absorbing resin (D) is produced by aqueous polymerization performed using a monomer (A) aqueous solution that has a concentration in the range of from 30% by weight to a saturation contains concentration and (i) water-soluble ethylene-based unsaturated monomer (A), (ii) 0.005 mol% to 5 mol% of cross-linking agent (B) with respect to (iii) 0.001 mol% to 1 mol% monomer (A), and water-soluble chain transfer agent (C) with respect to monomer (A).
- 3. A production method as set forth one of Claims 1 and 2, wherein water-soluble chain transfer agent (C) is hypophosphite.
- 4. A production method as set forth in one of Claims 1 and 2, wherein an amount of cross-linking agent (B) to be used is 0.02 mol% to 1 mol% with respect to water-soluble ethylene-based unsaturated monomer (A).
 - 5. A production method as set forth in one of Claims

- 1 and 2, wherein the concentration of the water-soluble ethylene-based unsaturated monomer (A) aqueous solution is in the range of from 35% by weight to the saturation concentration.
- 6. A production method as set forth in one of Claims 1 and 2, wherein water-soluble ethylene-based unsaturated monomer (A) contains at least 50% of (i) acrylic acid and (ii) acrylic acid alkali metal salt and/or ammonium acid.
- 7. A production method as set forth in Claims 1 and 2, wherein the aqueous polymerization is performed using the water-soluble ethylene-based unsaturated monomer (A) aqueous solution in a reactor vessel having a rotation stir blade.
- 8. A production method as set forth in Claim 7, wherein the reactor vessel has a plurality of rotation stir blades.
- 9. A production method as set forth in Claim 8, wherein the reactor vessel having the plurality of rotation stir blades is a two-screwed kneader.
 - 10. A production method as set forth in Claim 9,

wherein 0.04 mol% to 0.4 mol% of cross-linking agent (B) is used with respect to water-soluble ethylene-based unsaturated monomer (A).

- 11. A production method as set forth in Claim 2, wherein, in (i) 0 parts by weight to 20 parts by weight of water and (ii) 0 parts by weight to 20 parts by weight of hydrophilic organic solvent (G), 0.005 parts by weight to 5 parts by weight of at least one type of hydrophilic cross-linking agent (E) selected from compounds (E-1) containing two or more functional groups reactive with a carboxyl group and/or multivalent metal salt (E-2) is mixed and heated at a temperature of 40°C to 250°C with respect to 100 parts by weight of water-absorbing resin (D).
- 12. A production method as set forth in Claim 11, wherein hydrophilic cross-linking agent (E) is a compound (E-1) or compounds (E-1).
- 13. A production method as set forth in Claim 12, wherein the compounds (E-1) are one type or more than one type of compound selected from a polyhydroxy alcohol, a multivalent glycidylether compound, a multivalent oxazoline compound, or a multivalent amine compound.

- 14. A production method as set forth in Claim 11, wherein 0.5 parts by weight to 10 parts by weight of water is used with respect to 100 parts by weight of water-absorbing resin (D).
- 15. A production method as set forth in Claim 11, wherein 0 parts by weight to 8 parts by weight of hydrophilic organic solvent (G) is used with respect to 100 parts by weight of water-absorbing resin (D).
- 16. A production method as set forth in Claim 11, wherein the heating is performed at a temperature of 90°C to 220°C.
- Detailed description of the invention
 [Industrially applicable field]

The present invention relates to a production method of a water-absorbing resin excellent in durability. Specifically, the present invention relates to a production method of a water-absorbing resin that: has excellent absorbency; has excellent absorption rate; has excellent durability after having swelled; can form a swelled gel that is less sticky; and has excellent liquid permeability.

Such water-absorbing resin can be produced inexpensively and easily. Furthermore, the water-absorbing resin is excellent in safety and absorption

power. Therefore, the water-absorbing resin can be widely used in sanitary materials (e.g., sanitary napkins, paper diapers), in water supplying agents for agricultural activities, horticultural activities, or greenery, or in any other materials of absorbing products.

[Prior art]

In recent years, a water-absorbing resin that absorbs water of several tens to hundreds times more amount of a weight of the water-absorbing resin has been developed. Such water-absorbing resin has been widely used in various fields including, for example, a field of sanitary materials (e.g., paper diapers, sanitary napkins), a field of agriculture and forestry, and a field of civil engineering.

As a main raw material of the water-absorbing resin, example the following materials are known: containing partially-neutralized cross-linked polymer polyacrylic acid (Japanese Unexamined Patent Publication No. 55-84304); a hydrolysate which is a starch-acrylic graft polymer (Japanese Examined Patent Publication No. 49-43395); a saponificated vinyl acetate-acrylic ester copolymer (Japanese Unexamined Patent Publication No. cross-linked hydrolyzed 52-14689); acrylonitril hydrolyzed acrylamide copolymer copolymer or (Japanese Examined Patent Publication No. 53-15959); and cross-linked products thereof.

Such water-absorbing resin is demanded to exhibit,

for example, high absorbency and fast absorption rate when contacting with an aqueous liquid, and excellent suction power for absorbing liquid from a material containing the aqueous liquid.

However, depending upon the usage of water-absorbing resin, especially durability and temporal stability (stability in terms of changes over time) of the swelled gel, in addition to the properties mentioned above, become necessary. For example, in the case where a conventional water-absorbing resin is used in sanitary materials (e.g., paper diapers), there arises a problem in that a swelled gel of the water-absorbing resin having absorbed urine is deteriorated or decomposed as time passes by. Further, in the case where a conventional water-absorbing resin is used for a long period of time in agricultural or horticultural activities, there also arises a problem in that the swelled gel of the water-absorbing resin is deteriorated or decomposed.

Examples of known methods for preventing deterioration and decomposition of the swelled gel of the water-absorbing resin include: a method wherein an oxygen-containing reducing inorganic salt or a radical chain inhibitor is contained a water-absorbing resin (Japanese Unexamined Patent Publications Nos. 63-118375 and 63-152667); a method wherein an oxidant is contained in a water-absorbing resin (Japanese

Unexamined Patent Publication No. 63-153060); or method wherein a reductant containing sulfur (Japanese contained water-absorbing resin in а Unexamined Patent Publication No. 63-272349). These methods, however, require to add an additive in order to prevent the water-absorbing resin from deteriorating. From the point of view of safety, it is not preferable to add another additives, considering that the water-absorbing resin is to be used in sanitary materials or the like.

In this connection, a greater amount of cross-linking agent may be used to increase a cross-linking density of the water-absorbing resin so that the strength of the gel is enhanced and therefore the durability is improved. However, if a greater amount of cross-linking agent that is sufficient for the water-absorbing resin to have enough durability is used, the absorbency decreases significantly. As such, currently, water-absorbing resin that is excellent in safety, high in absorbency, and excellent in durability has not been realized, yet.

Further, in the case where the water-absorbing resin is used in, for example, diapers, there arise additional problems of stickiness of the swelled gel and reduction in the liquid permeability, in addition to the problem of durability mentioned above. The water-absorbing resin contains a water-soluble portion (the portion will be referred to as a water-soluble content). Because of the

presence of the water-soluble content, the swelled gel having absorbed water may become sticky. Further, in the case where the water-absorbing resin is used in absorbing products such as diapers, the liquid permeability may be reduced due to the stickiness. Therefore, when additional urine is supplied, the urine may be leaked. Normally, the amount of water-soluble content and the absorbency are correlation. In order reduce in positive to the water-soluble content, the cross-linking density of the water-absorbing resin needs to be increased. Therefore, if the water-absorbing resin has less water-soluble content, the absorbency also becomes lower.

However, it has been publicly known that the more proportion of the cross-linking agent water-absorbing resin is, the more improved the durability would be. However, there is a problem in that the absorbency decreases as the amount of cross-linking agent increases. Further, there has been disclosed a technique in which a chain transfer agent is used in production of water-absorbing resins to improve the absorbency (USP4698404). In this case, however, only few increase in the absorbency is observed for human urine, while the absorbency assuredly improves for water and physiological saline.

[Problem to be solved by the invention]

The present invention is in view of the current

situation described above, and has as an object to provide a production method of a water-absorbing resin excellent in durability.

Another object of the present invention is to provide a production method of a water-absorbing resin that (i) exhibits high absorbency with respect to a physiological saline and especially to a human urine, (ii) exhibits excellent durability when used in disposable diapers, (iii) prevents diapers from exuding liquid absorbed therein, (iv) can form a gel that is less sticky, and (v) exhibits excellent liquid permeability.

[Means for solving problem and effect]

In order to solve the above problems, the inventors have diligently researched, and finally found the followings:

(1) By performing aqueous polymerization, using a particular amount of cross-linking agent (B), on a water-soluble ethylene-based unsaturated monomer (A) aqueous solution that has a concentration in a particular range and contains water-soluble chain transfer agent (C), desirable water-absorbing resin (D) whose (i) durability has improved, (ii) can form gel that is less sticky, and (iii) liquid permeability has improved, while maintaining high absorbency, due to reduced molecular weight of the water-soluble content can be produced with good productivity.

(2) Further, by cross-linking, using hydrophilic cross-linking agent (E), the vicinity of the surface of the water-absorbing resin (D) produced by production method (1) described above, water-absorbing resin (F) that exhibits (a) better water absorbency, (b) better durability, (c) less gel stickiness, (d) better liquid permeability, and (e) better water absorption property, while maintaining high absorbency, can be produced.

In other words, the present invention relates to:

a production method of a water-absorbing resin (D) excellent in durability, which method is adapted so that the method includes performing aqueous polymerization using a monomer (A) aqueous solution that has a concentration in the range of from 30% by weight to a saturation concentration and contains (i) water-soluble ethylene-based unsaturated monomer (A), (ii) 0.005 mol% to 5 mol% of cross-linking agent (B) with respect to and monomer (A), and (iii) 0.001 mol% to 1 mol% of water-soluble chain transfer agent (C) with respect to monomer (A); and

a production method of a water-absorbing resin (D) excellent in durability, which method is adapted so that the method includes performing cross-linking in the vicinity of a surface of water-absorbing resin (D) is cross-linked by a hydrophilic cross-linking agent that is reactive with a functional group contained in

water-absorbing resin (D), which water-absorbing resin (D) is produced by aqueous polymerization performed using a monomer (A) aqueous solution that has a concentration in the range of from 30% by weight to a saturation contains and (i) water-soluble concentration ethylene-based unsaturated monomer (A), (ii) 0.005 mol% to 5 mol% of cross-linking agent (B) with respect to and (iii) 0.001 mol% to 1 mol% of monomer (A), and water-soluble chain transfer agent (C) with respect to monomer (A).

The following specifically describes the present invention.

Water-soluble ethylene-based unsaturated monomer (A) (the monomer will be referred to as monomer (A)) of the present invention contains a functional group. Examples of monomer (A) include acrylic acid, methacrylic acid, maleic acid, fumaric acid, crotonic acid, itaconic acid, acid, vinylbenzenesulfonic 2-(meth)acrylamide-2-methylpropanesulfonic acid, 2-(meth)acryloylethanesulfonic acid, and their alkali 2-(meth)acryloylpropanesulfonic acid, acrylamide, metal salts and ammonium salts: and 2-hydroxyethyl(meth)acrylate, methacrylamide, methoxypolyethyleneglycol(meth)acrylate, N, N-dimethylaminoethyl(meth)acrylate,

N, N-diethylaminopropyl(meth)acrylate,

N,N-diethylaminopropyl(meth)acrylamide, and their quaternary salts. At least one of the above may be selected and used.

In view of properties and costs of water-absorbing resin that is produced, it is preferable that acrylic acid, among monomers (A) listed above, be used as a main content. In this case, acrylic acid and its alkali metal salt and/or ammonium salt is contained, preferably, at a percentage of 50% by weight or greater, and more preferably, at a percentage of 75% by weight or greater with respect to monomer (A).

Further, in order to produce a water-absorbing resin excellent in durability, in the present invention, it is necessary to use a particular amount of cross-linking agent (B) that contains two or more polymerizable unsaturated groups and/or a reactive functional group per molecule. As cross-linking agent (B), for example a compound containing two more polymerizable or unsaturated group per molecule may be used. Examples thereof include. N, N'-methylenebisacrylamide, (poly)ethyleneglycoldi(meth)acrylate,

(poly)propyleneglycoldi(meth)acrylate,

glycerictri(meth)acrylate,

glycericacrylatemethacrylate, multivalent metal salts of (meth)acrylic acid, trimethylolpropanetri(meth)acrylate, triallylamine, triallylcyanurate, triallylisocyanurate, and

triallylphosphate and the like. Further, examples cross-linking agent (B) containing reactive functional groups in the case where monomer (A) contains a carboxyl group include: polyhydric alcohols such as ethylene diethylene glycol, triethylene glycol, glycol; polyethyleneglycol, propylene glycol, dipropylene glycol, polypropylene glycol, and glycerin; multivalent glycidyl compounds such as (poly)ethylene glycoldiglycidylether, and glycerolpolyglycidylether; multivalent amines such as polyethyleneimine; multivalent ethylenediamine, and isocyanate compounds; multivalent metal salts and the Further, examples of cross-linking agent containing both a polymerizable unsaturated group and a molecule include functional per reactive group glycidyl(meth)acrylate and N-methylol(meth)acrylamide. From the points of view of durability and water absorbency of the water-absorbing resin that is produced, it is especially preferable to use at least one of the compounds containing more than one polymerizable molecular, among unsaturated groups in а cross-linking agents (B).

The amount of cross-linking agent (B) to be used in the present invention is 2 to 10 times greater in mol, or more preferably 4 to 10 times greater in mol than the amount of the cross-linking agent used in the case where the polymerization is performed under the same conditions, except that water-soluble chain transfer agent (C) is not used to obtain a target water-absorbency. Specifically, the amount of cross-linking agent (B) to be used is 0.005 mol% to 5 mol% with respect to monomer (A). If less than 0.005 mol% of cross-linking agent (B) is used, a water-absorbing resin with high absorbency is produced. water-absorbing resin inferior However, the is durability, contains many water-soluble contents, and has a higher molecular weight. This causes the gel of the water-absorbing resin to be stickier, and the liquid permeability to be degraded. On the other hand, if more than 5 mol% of cross-linking agent (B) is used, the absorbency becomes extremely low. Further, preferable that the amount of cross-linking agent to be used be in the range of from 0.02 mol% to 1 mol%, more preferably in the range of from 0.04 mol% to 0.4 mol%, and even more preferably in the range of from 0.08 mol% to 0.2 mol%, although it depends upon the amount of water-soluble chain transfer agent (C) to be used. The amount of water-soluble chain transfer agent (C) will be described later.

Further, in addition to the above method utilizing cross-linking agent (B), a method in which graft polymerization is performed to form a cross-link may be carried out simultaneously. For example, the monomer (A) aqueous solution may be polymerized in the presence of a

hydrophilic polymer (e.g., cellulose, starch, polyvinyl alcohol), so that a cross-link is formed as a result of the graft polymerization. It is preferable that the water-soluble macromolecule be used in the range of from 1% by weight to 50% by weight with respect to monomer (A).

In the present invention, it is necessary to use a particular amount of water-soluble chain transfer agent (C).

According to the present invention, (i) a particular amount of water-soluble chain transfer agent (C) is selected, and (ii) a large amount of cross-linking agent is used in polymerization to form a high cross-link, which amount is 2 to 100 greater in mol than that used in an ordinary production method. With such an arrangement, invention provides excellent the present an water-absorbing resin that (i) is highly excellent in durability, (ii) exhibits high absorbency not only to physiological saline but also to human urine, (iii) provides less negative influence (e.g., stickiness of gel, decrease in the liquid permeability) due to low molecular weight of the water-soluble content can be produced.

The water-soluble chain transfer agent (C) for use in the present invention is not particularly limited, provided that water-soluble chain transfer agent (C) is soluble in water or in a water-soluble ethylene-based unsaturated monomer. The water-soluble chain transfer agent (C) may be a thiol, a thiol acid, a secondary alcohol, an amine, a hypophosphite, or the like. Specific examples of the transfer (C) water-soluble chain agent include: mercaptoethanol, mercaptopropanol, dodecyl mercaptan, thioglycolic acid, thiomalic acid, 3-mercaptopropionic acid, isopropanol, sodium hypophosfite, formic acid, and their salts. One of, or two or more of the water-soluble chain transfer agents (C) are selected and used. Because of their effect, it is preferable that the water-soluble chain transfer agent (C) be hypophosphite such as sodium hypophosfite.

An amount of the water-soluble transfer agent (C) to add is, with respect to monomer (A), in the range of from 0.001 mol% to 1 mol%, preferably in the range of from 0.005 mol% to 0.3 mol%, even though it depends on the type and amount of internal cross-linking agent (B) and the concentration of monomer (A) aqueous solution. In the case where the amount is less than 0.001 mol%, the amount of cross-linking agent (B) to be used in the present invention is not preferable because the cross-linking density would become high the absorbency would become too low. Further, it is not preferable to use more than 1 mol% of cross-linking agent (B) because the water-soluble content would increase and the durability would decrease.

The concentration of monomer (A) aqueous solution used in the present invention is in the range of from 30% by weight to the saturation concentration. It is more preferable that the concentration be in the range of from 35% by weight to the saturation concentration. If the concentration is below 30% by weight, the productivity per unit-reaction-volume decreases. Moreover, the drying step and therefore the productivity takes longer time, decreases. Therefore, from industrial view, the concentration below 30% by weight is not preferable. In polymerization methods, conventional polymerization is performed at a concentration close to the saturation concentration in order to improve the reactions productivity, unnecessary such self-cross-linking occur, and the absorbency is decreased. Therefore, the amount of cross-linking agent (B) that can be used in the polymerization is limited, and thus only a water-absorbing resin having poor durability is produced. However, with the method of the present invention, self-cross-linking reaction is prevented by choosing the amount of water-soluble chain transfer agent (C) to be used. This makes it possible to increase the amount of cross-linking agent (B) to be used, and therefore a water-absorbing resin that is excellent in durability and high absorbency can be produced at concentration at good productivity.

Further, if necessary, a thickener may be used in the monomer (A) aqueous solution. Examples of the thickener include: polyvinylpyrrolidone; polyacrylamide; methyl cellulose; and hydroxyethyl cellulose and the like.

In the present invention, among publicly-known polymerization techniques (e.g., aqueous polymerization, suspension polymerization, precipitate reverse-phased polymerization, bulk polymerization, polymerization by activation energy such as ultraviolet ray or electron ray), aqueous polymerization is employed for polymerizing using the monomer (A) aqueous solution to produce a water-absorbing resin that has excellent properties and productivity, and requires less costs. Exemplary methods of aqueous polymerization include: casting polymerization cast (Japanese Examined Patent performed in (Tokukosho 48-42466)); No. 48-42466 Publication polymerization performed using a belt conveyer (Japanese Unexamined Patent Publication No. 58-49714 (Tokukaisho 58-49714)); and polymerization performed in, for example, a kneader having a stir blade that can comminute hydrate gel polymers (Japanese Unexamined Patent Publication No. 57-34101 (Tokukaisho 57-34101)).

In performing the aqueous polymerization, it is preferable that (i) chain transfer reaction take place uniformly with water-soluble chain transfer agent (C) and (ii) the cross-linking reaction occurs uniformly with

(B). In order to produce agent cross-linking water-absorbing resin having better properties, it is generated during heat preferable that the polymerization be removed uniformly. In order to do so, it is preferable that, instead of the polymerization method in which the polymerized gel is kept as one united body, the polymerized gel of the reaction system be stirred from the beginning to the end of the polymerization, or sometimes throughout polymerization or the polymerization so that the heat generated during the polymerization be removed uniformly, instead polymerization method in which the polymerized gel is unified. It is thus preferable that the polymerization reaction be performed in a reactor vessel having a rotation stir blade. The reactor vessel having a rotation stir blade is not particularly limited, but a reactor vessel that can stir the polymerized gel with greater power is preferable. An exemplary reactor vessel is a vessel that shears the by the rotation blade, which stir polymerized gel generated as the polymerization polymerized gel is proceeds. Further, in order to increase the stirring power, it is preferable that a plurality of rotation stir blades be provided. Examples of the reactor vessel include a single-screwed mix-kneader, a single-screwed extruder, a two-screwed kneader, and a three-screwed kneader. Further, by using the two-screwed kneader, polymerized

gel can be fragmented and stirred evenly throughout the polymerization, and the heat generated during the polymerization can be removed uniformly. As a result, a water-absorbing resin having excellent properties can be produced. Therefore, it is preferable to use a two-screwed kneader.

In the present invention, the radical polymerization initiator to be used in the aqueous polymerization is not particularly limited, as long as the radical polymerization initiator is water-soluble. For example, the followings may be used as the radical polymerization initiator: persulfates such as potassium persulfate, ammonium persulfate, sodium persulfate and the like; hydroperoxides such as t-butylhydroperoxide, peroxide, hydrogen cumenehydroperoxide and the like; azo compounds such as 2,2'-azobis(2-amizinopropane)dihydrochloride) and the like; secondary cerium salt, permanganate, and the like. properties of view of, example, (i) for water-absorbing resin to be produced and (ii) safety of decomposition products, it is preferable that the radical polymerization initiator be one or more of persulfate, hydroperoxide, and azo compounds.

Further, in the case where the radical polymerization initiator is an oxidizing radical polymerization initiator, reductants may be combined to be used as a redox type initiator. Examples of the reductants include: sulfites

(hydrogen salt) such as sodium sulfite, sodium hydrogen sulfite and the like; thisulfate such as sodium thiosulfate and the like; dithionite; metal salt such as copper sulfate, iron sulfate and the like; organic reductants such as b-ascorbic acid and the like; aniline; and amines such as monoethanolamine and the like.

Although a wide range of the amount of the radical polymerization initiator may be used, normally it is preferable that the range of the amount of the radical polymerization initiator be 0.001 mol% to 2 mol% with respect to monomer (A), or more preferably 0.01 mol% to 0.5 mol% with respect to monomer (A). If less than 0.001 mol% of radical polymerization initiator is used, the induction period becomes longer and the amount of the easily increases. Further, in residual monomer conventional polymerization method, if the amount of the polymerization initiator is increased in order to (a) reduce the amount of the residual monomers or (b) shorten the polymerization period, and the induction period unnecessary reactions such as self-cross-linking may occur, thereby degrading the absorbency. Thus, the amount of initiator has been limited. On the contrary, with the present invention, these disadvantages are solved, and the water-absorbing resin having excellent properties is produced even when the amount of initiator is increased. If, however, more than 2 mol% of radical polymerization

initiator is used, not only expected effects from the increased amount cannot be achieved, but also it becomes difficult to control the polymerization reaction. Accordingly, it is not preferable to use more than 2 mol% of radical polymerization initiator.

The water-absorbing resin produced by the present invention may be polymerized at high concentration, and the water-absorbing resin may be dried with the heat polymerization while during the generated polymerized. Depending upon the proportion of water contained after performance of the polymerization, the hydrate gel produced may be dried further and then used For the drying, water-absorbing resin. in the drying methods may be employed. publicly-known Exemplary methods thereof include: azeotropy dehydration in an organic solvent; drying with a mechanical draft oven; drying with a reduced-pressure dryer; drying with a microwave dryer; drying with an infrared dryer; and drying with a belt having heated to a predetermined temperature or a drum dryer having heated to predetermined temperature. It is preferable that the polymerized hydrate gel be dried by one of the above methods at 80°C or above. It is more preferable that the hydrate gel be dried at 80°C to 230°C. In view of productivity, it is not preferable to dry the hydrate gel at a temperature below 80°C because it consumes too much time. Careful attention is required in drying the hydrate gel at 230°C or above because the water-absorbing resin may be deteriorated at such high temperature.

Further, the water-absorbing resin produced by the polymerization and the drying is pulverized and/or classified, if necessary, before being used.

Further, the present invention also provides production method of water-absorbing resin (F). In the production method, the water-absorbing resin (F) is produced by cross-linking, with particular hydrophilic cross-linking agent (E), in the vicinity of the surface of water-absorbing resin (D) produced by the production method described above. Water-absorbing resin (D) produced by the production method of the present invention has significantly improved water absorbency, compared with conventional water-absorbing Further, water-absorbing resin (F), on which cross-linking was performed in the vicinity of the surface, has better durability and water absorbency than water-absorbing resin (D), on which cross-linking has not been performed in the vicinity of the surface.

Hydrophilic cross-linking agent (E) used in the present invention is compound (E-1) containing more than one functional group per molecule and/or multivalent metal salt (E-2) containing more than one functional group per molecule, which functional group is reactive

with a carboxyl group. Examples of compounds (E-1) in the case where water-absorbing resin (D) contains a carboxyl group include: polyhydroxy alcohols such as ethylene glycol, diethylene glycol, triethylene glycol, tetraethylene glycol, polyethyleneglycol, 1,4-butanediol, 1,6-hexanediol, neopentylglycol, 1,5-pentanediol, polyglycerin, glycerin, glycol, propylene trimethylolpropane, pentaerythritol, sorbitol, polyvinyl the like; ethylene glycoldiglycidylether; and glycidylether compounds such as multivalent ethyleneglycoldiglycidylether,

polyethyleneglycoldiglycidylether, glycerolpolyglycidylether, diglycerolpolyglycidylether, polyglycerolpolyglycidylether, glycoldiglydiglycidylether, polypropylene propylene glycoldiglycidylether and the like; multivalent amine compounds such as ethylenediamine, diethylenetriamine, like: triethylenetetramine, polyethyleneimine and the multivalent oxazoline compounds such as 1,2-ethylenebisoxazoline, polyisopropenyloxazoline the like; haloepoxy compounds such as epichlorohydrin and the like; multivalent aziridine compounds; and multivalent isocyanate compounds; and the like. Examples of multivalent metal salt (E-2) in the case where water-absorbing resin (D) contains a carboxyl group zinc, hydroxide of calcium, hydroxide of include: magnesium, hydroxide of aluminum, hydroxide of

hydroxide of iron, hydroxide of zirconium, chloride of zinc, chloride of calcium, chloride of magnesium, chloride of aluminum, chloride of iron, and chloride of zirconium, and the like. It is preferable that one kind or more than one kind of the above be used. It is preferable to use compound (E-1) as an essential component. In view of surface cross-linking effects, it is especially preferable that a polyhydric alcohol, a multivalent glycidyl compound, a multivalent amine be used hydrophilic as Further, a cross-linking agent (E). combination of compound (E-1) and multivalent metal salt (E-2) may be used as hydrophilic cross-linking agent (E) to improve combined properties.

The amount of hydrophilic cross-linking agent (E) used in the present invention is 0.005 parts by weight to 5 parts by weight with respect to 100 parts by weight of water-absorbing resin (D) produced by the production method described above. It is preferable that the amount be in the range of from 0.01 parts by weight to 1 part by weight with respect to 100 parts by weight water-absorbing resin (D) produced by the production method described above. If the amount is within this excellent water-absorbing resin (F) having range, properties and cross-linked surface can be produced. If more than 5 parts by weight of hydrophilic cross-linking agent (E) is used, it is not only uneconomical, but also

may cause unreacted hydrophilic cross-linking agent (E) to remain in water-absorbing resin (F) that has been amount of hydrophilic Moreover, this produced. cross-linking agent (E) is excess for achieving desired cross-linking effects, reducing the water absorbency of water-absorbing resin (F) that is produced. Therefore, it is not preferable to use more than 5 parts by weight of hydrophilic cross-linking agent (E). Further, in the case where less than 0.005 parts by weight of hydrophilic cross-linking agent (E) is used, it is difficult to achieve the effects of the present invention.

In the present invention, water and/or hydrophilic organic solvent (G) may be used in mixing water-absorbing resin (D) and hydrophilic cross-linking agent (E).

In the present invention, the amount of water to be used is 0 part to 20 parts by weight with respect to 100 parts by weight of water-absorbing resin (D). It is preferable that the amount be in the range of from 0.5 parts by weight to 10 parts by weight.

Examples of hydrophilic organic solvent (G) include: lower alcohols such as methanol, ethanol, n-propanol, iso-propanol, n-butanol, iso-butanol, t-butanol and the acetone, methylethylketone, ketones such as like; methylbutylketone and the like; ethers such as dioxane tetrahydrofuran and the like: amides such N, N-dimethylformamide and the like; and sulfoxides such as dimethylsulfoxide and the like. The amount of water-absorbing resin (D) to be used is in the range of from 0 parts by weight to 20 parts by weight with respect to 100 parts by weight of water-absorbing resin (D). It is preferable that the amount be in the range of from 0 parts by weight to 8 parts by weight.

The followings are exemplary methods, according to the present invention, of (i) mixing water-absorbing resin (D) and hydrophilic cross-linking agent (E) and (ii) cross-linking in the vicinity of the surface of the water-absorbing resin:

- (i) Hydrophilic cross-linking agent (E) and, if necessary, a mixed solution of water (water vapor) and/or hydrophilic organic solvent (G) may be added to water-absorbing resin (D) by spraying or dropping;
- (ii) Water-absorbing resin (D) is dispersed and suspended in a hydrophobic organic solvent that is inactive in polymerization, and hydrophilic cross-linking agent (E) and, if necessary, water and/or hydrophilic organic solvent (G) are added while being stirred; and
- (iii) water-absorbing resin (D) is dispersed in a mixed solvent of water and hydrophilic organic solvent (G), and hydrophilic cross-linking agent (E) is added.

In case (ii), it is preferable that hydrophilic cross-linking agent (E) be emulsified, using a particular surfactant, into a hydrophobic organic solvent and be

suspended in advance, and then the emulsified solution be added to a solution in which water-absorbing resin (D) has been dispersed and suspended.

As described above, an ordinary heating machine or heating oven may be used to perform a heating treatment on a mixture of (i) water-absorbing resin (D) produced by the method of the present invention, (ii) hydrophilic cross-linking agent (E), and, if necessary, water and/or hydrophilic organic solvent (G). Examples of the heating machine and the heating oven include: a trench type mixing dryer; a rotary type dryer; a disk type dryer; a kneading dryer; a fluidized bed type dryer; an air-flow type dryer; an infrared dryer; a dielectric heating dryer; and the like. Further, hydrophilic cross-linking agent (E) may be added, in an organic solvent, to water-absorbing resin (D) by method (ii), and thereafter heated to react in the organic solvent.

It is preferable that the heat treatment be performed at the heating treatment in the range of from 40°C to 250°C, more preferably in the range of from 90°C to 220°C, although it depends upon which kind of hydrophilic cross-linking agent (E) is used. If the heating treatment is performed at a temperature below 40°C, not only it takes time to react and therefore the productivity decreases, but also a part of hydrophilic cross-linking agent (E) may not react and may remain in water-absorbing resin (F) that is

produced. Therefore, it is not preferable that the heating treatment be performed at a temperature below 40°C. On the other hand, if the heating treatment is performed at a temperature above 250°C, deterioration by the heat may occur in some kinds of water-absorbing resin (D). Therefore, careful attention is required.

Note that water-absorbing resin (F) whose surface is cross-linked or water-absorbing resin (D) whose surface is not cross-linked may be pulverized and agglomerated, if necessary.

[Effect of the invention]

The water-absorbing resin produced by the method of the present invention has the following advantages, which could not be achieved by the conventional methods: excellent durability of swelled gel; high absorbency with respect to physiological saline and especially to human urine; low molecular weight of the water-soluble content; reduced stickiness of swelled significantly significantly improved liquid permeability; and excellent safety. According to the method of the present invention, such excellent water-absorbing resin can be easily produced by performing aqueous polymerization, in the presence of a particular amount of cross-linking agent (B) and a particular amount of water-soluble chain transfer agent (C), on water-soluble ethylene-based unsaturated Furthermore, by cross-linking, monomer (A).

particular hydrophilic cross-linking agent (E), in the vicinity of the surface of water-absorbing resin (D) that has been produced, water-absorbing resin (F) having significantly improved absorption rate and durability can be produced. Water-absorbing resin (D) and water-absorbing resin (F) that have excellent water absorbency can be produced inexpensively. This could not be achieved by a conventional water-absorbing resin. Therefore, water-absorbing resin (D) and water-absorbing resin (F) may be widely used in various fields including sanitary material, foods, civil engineering, agriculture.

As described above, the water-absorbing resin produced by the production method of the present invention has the following advantages.

- (1) The swelled gel has excellent durability because, in the present invention, the high absorbency is maintained even when a large amount of cross-linking agent is used. In contrast, conventional swelled gels have had poor durability because polymerization is performed using a reduced amount of cross-linking agent in order to produce a water-absorbing resin having high absorbency.
- (2) The absorbency with respect to human urine is high. This could not be achieved by the conventional water-absorbing resins.
- (3) The water-absorbing resins can be produced at high productivity because even when the monomer is

polymerized at a high concentration close to the saturation concentration, a water-absorbing resin having excellent properties can be produced without causing unnecessary reactions such as self-cross-linking. Therefore, the polymerization can be performed at a high concentration.

- The water-absorbing resin of the invention has high absorbency, and the water-soluble content associated therewith does not provide negative influences (e.g., stickiness of swelled gel due content, reduction in the liquid water-soluble permeability) because the molecular weight of the water-soluble content is small.
- (5) By performing the cross-linking in the vicinity of the surface, (i) the water absorbency can be better than the absorbency achieved by a conventional surface cross-linking of water-absorbing resin, and (ii) the durability and the absorption rate can be improved.
- (6) A small amount of residual monomer is used, because high absorbency can be achieved even when an increased amount of polymerization initiator is used. In contrast, conventionally, if the amount of polymerization initiator is increased in order to reduce the residual monomer or shorten the induction period and polymerization period, unnecessary reaction such as self-cross-linking is generated. Therefore, high absorbency

could not be achieved.

(Examples)

The following describes the present invention, with reference to Examples. The scope of the present invention, however, is not limited to the Examples. Note that the properties of the water-absorbing resin in the Examples indicate the values measured by the testing methods described later. Further, the term "part(s)" means "part(s) by weight".

(1) Absorbency of physiological saline

In a beaker containing 150 ml of 0.9% by weight chloride sodium aqueous solution, 1.0 of water-absorbing resin was soaked and stirred slowly using a magnetic stirrer. After six hours had passed, the swelled gel was filtered by a wire net, and the weight of the which swelled gel from water had been adequately was measured. Then, the absorbency was calculated using the formula below.

absorbency(g/g) = weight of swelled gel/weight of water-absorbing resin

(2) Absorbency of human urine

The absorbency of human urine was measured under the same conditions, except that sample human urine of 10 adult males were used instead of the physiological saline used in the case (1).

(3) Water-soluble content

In 1000 ml of deionized water, 0.5 g of water-absorbing resin was dispersed. After 12 hours had passed, this was filtered by a paper filter, and the solid content in the filtrate was measured. Then, the water-soluble content was calculated using the formula below.

water-soluble content (%) =

weight of filtrate (g) x solid content in filtrate (%)

0.5 g

(4) Molecular weight of water-soluble content

By using, as standard, various polysodium acrylic acids whose molecular weights had already been calculated, the molecular weight of the water-soluble content obtained in item (3) was worked out by using gel permeation chromatography.

(5) Tolerance of swelled gel

A commercially-available diaper for children (weight: 72 g) formed of nonwoven fabric, cotton-like pulp, water-absorbing paper, and water-proof film is cut into a half, and 2.5 g of polymer was evenly dispersed between the cotton-like pulp and the water-absorbing paper, and 120 ml of adult human urine was added. Then, the diaper

was left at 37°C. Thereafter, the diaper was opened to observe the state of the swelled gel after (i) 6 hours, (ii) 12 hours, and (iii) 18 hours. The degree of deterioration was evaluated using three categories: good, average, and poor.

Good : shape of swelled gel is maintained

Average : shape of swelled gel is partially collapsed

Poor : shape of swelled gel is collapsed,

and the swelled gel is muddy

(6) Amount of exudation of absorbed liquid

On the nonwoven fabric of the diapers for children after 18 hours, which diapers had been used in the examination of durability of the swelled gel, 10 sheets of 23-by-23-cm paper towels folded into two were overlaid. Then, a pressure of 40 g/cm² was applied for one minute. Thereafter, the amount of urine returned to the paper towels was measured.

(7) Stickiness of swelled gel

The stickiness (feeling of dryness) of the swelled gel whose absorbency had been calculated in (1) was determined by hand-touching. The feeling of dryness was evaluated using three categories: good, average, and poor.

Good : swelled gel is very dry,

providing feeling of dryness

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Average : swelled gel is partially sticky

Poor : swelled gel is sticky,

and hand becomes slimy

(8) Liquid permeability of swelled gel

As illustrated in Figure 1, in a plate 1 having an inner diameter of 53 mm, 1.0 g of water-absorbing resin was placed and 10 ml of human urine was poured therein, thereby obtaining swelled gel 2. On the swelled gel 2, a paper towel 3 having a diameter of 53 mm was placed. Further, a test tube 4 made of acrylic resin, provided with a protruding cylinder, and having doughnut-shaped was placed at the center thereof, as illustrated in Figure 1. After this was left at a room temperature for one hour, 6 ml of human urine was poured from the inlet opening 5. The time taken for all of the human urine to be absorbed into the polymer was measured. The measured time is used as a parameter of the liquid permeability of the swelled gel.

(Example 1)

A monomer aqueous solution with the concentration of 37% and the neutralization ratio of 75% was produced using (i) 4380 g of 37% by weight aqueous solution containing 414 g of acrylic acid and sodium acrylic acid, (ii) 6.815 g (0.1 mol% with respect to monomer (A)) of trimethylolpropanetriacrylate, which functions as

cross-linking agent (B), (iii) 0.195 g (0.008 mol% with respect to monomer (A)) of sodium hypophosfite1hydrate, which functions as water-soluble chain transfer agent (C), and (iv) 670 g of ion-exchange water. Thereafter, nitrogen gas was blown to expel dissolved oxygen.

In a reactor vessel having a lid and a twin-arm type kneader (content volume of 10 liters) made stainless-steel and having a jacket and two sigma blades, monomer (A) aqueous solution was supplied and nitrogen gas was blown to fill a reaction system with nitrogen. Thereafter, the two sigma blades were rotated, while hot water of 35°C was streamed in the jacket to heat, 2.62 g of ammonium persulfate and 0.12 g of sodium hydrogen sulfite, both of which function as polymerization initiators, were added to start polymerization. At a peak of polymerization, hydrate gel polymer was fragmented to have a diameter of approximately 5 mm. Then, the stirring was continued, and 60 minutes after the beginning of the polymerization, the lid was moved to take out the gel. The fine particles of hydrate gel polymer that had been produced were (i) dispersed over a wire net having 50 meshes and (ii) dried with hot air of 170°C for 50 minutes. Then, the dried hydrate gel polymer was pulverized with a hammer type pulverizer, and filtered by a wire metal having 20 meshes. As a result, water-absorbing resin (1) that had filtered through the wire net having 20 meshes

was produced.

The properties of water-absorbing resin (1) were evaluated, and results of the evaluation are shown in Table 1.

(Example 2)

The same operations as in Example 1 were performed, except that 1.219 g (0.05 mol% with respect to monomer (A)) of water-soluble chain transfer agent (C) sodium hypophosfite1hydrate was used. As a result, water-absorbing resin (2) was produced.

The properties of water-absorbing resin (2) were evaluated, and results of the evaluation are shown in Table 1.

(Example 3)

The same operations as in Example 1 were performed, except that (i) 21.988 g (0.2 mol% with respect to monomer) of polyethyleneglycoldiacrylate (n=8) was used as cross-linking agent (B), and (ii) 2.44 g (0.1 mol% with respect to monomer (A)) of water-soluble chain transfer agent (C) sodium hypophosfite1hydrate was used. As a result, water-absorbing resin (3) was produced.

The properties of water-absorbing resin (3) were evaluated, and results of the evaluation are shown in Table 1.

(Example 4)

The same operations as in Example 1 were performed,

except that (i) 54.97 g (0.5 mol% with respect to monomer) of cross-linking agent (B) polyethyleneglycoldiacrylate(n=8) was used, and (ii) 9.76 g (0.4 mol% with respect to monomer (A)) of water-soluble chain transfer agent (C) sodium hypophosfite1hydrate was used. As a result, water-absorbing resin (4) was produced.

The properties of water-absorbing resin (4) were evaluated, and results of the evaluation are shown in Table 1.

(Example 5)

The same operations as in Example 1 were performed, except that (i) 2.152 g of N,N'-methylenebisacrylamide (0.06 mol% with respect to monomer (A)) was used in place of cross-linking agent (B), and (ii) 3.45 g of thiomalic acid (0.1 mol% with respect to monomer) was used in place of water-soluble chain transfer agent (C). As a result, water-absorbing resin (5) was produced.

The properties of water-absorbing resin (5) were evaluated, and results of the evaluation are shown in Table 1.

(Example 6)

The same operations as in Example 1 were performed, except that (i) the amount of ion-exchange water to be used was changed to 50 g, (ii) the monomer (A) aqueous solution with the concentration of 42% was used, (iii) 17.07 g (0.1 mol% with respect to monomer (A)) of

polyethyleneglycoldiacrylate(n=14) was used as cross-linking agent (B), and (iv) 0.69 g (0.02 mol% with respect to monomer (A)) of water-soluble chain transfer agent (C) thiomalic acid was used. As a result, water-absorbing resin (6) was produced.

The properties of water-absorbing resin (6) were evaluated, and results of the evaluation are shown in Table 1.

(Example 7)

Forty grams of corn starch and 600 g of ion-exchange water were placed in a reactor vessel provided with a stir-stick, a nitrogen gas blow tube, and a thermometer, stirred for one hour at 55°C, and thereafter cooled down to 30°C. To the starch aqueous solution, (i) 300 g of acrylic acid, (ii) 1.92 g (0.3 mol% with respect to monomer (A)) of N,N'-methylenebisacrylamide, which functions as cross-linking agent (B), (iii) 0.94 g (0.15 mol% with respect to monomer (A)) of thiomalic acid, which functions as water-soluble chain transfer agent (C), were solved.

As a result, a monomer aqueous solution with the concentration of 33% and the neutralization ratio of 0% was produced.

The monomer aqueous solution was adjusted to 35°C, and 0.20 g of sodium persulfate, which functions as the polymerization initiator, and 0.04 g of l-ascorbic acid were added and then stirred for three hours to be polymerized.

To a hydrate gel polymer, 389 g of 30% by weight caustic soda aqueous solution was added so that the neutralization ratio is adjusted to 70%. Then, it was dried and pulverized in the same manner as in Example 1. As a result, water-absorbing resin (7) was produced.

The properties of water-absorbing resin (7) were evaluated, and results of the evaluation are shown in Table 1.

(Example 8)

To 72 g of acrylic acid, 22.2 g of deionized water was added. A mixture produced thereby was used as neutralizer, and (i) 49.5 g of potassium hydroxide with the purity of 85%, (ii) 0.1 g (0.065 mol% with respect to N, N'-methylenebisacrylamide, which monomer (A)) οf functions as cross-linking agent (B), 0.225 g (0.15 mol% with respect to monomer (A)) of thiomalic acid, which functions as water-soluble chain transfer agent (C), were added sequentially. As a result, a monomer aqueous solution with the concentration of 70% and the neutralization ratio of 75% was produced.

This monomer aqueous solution was kept at 70°C in a nitrogen air stream with a thickness of approximately 5 mm. Then, 0.1 g of ammonium persulfate and 0.02 g of sodium hydrogen sulfite were added, and polymerization was performed. The polymerization started immediately. After 10 minutes had passed, a polymerized gel that was

almost dried was taken out, dried, and pulverized, in the same manner as in Example 1. As a result, water-absorbing resin (8) was produced.

The properties of water-absorbing resin (8) were evaluated, and results of the evaluation are shown in Table 1.

(Example 9)

To 100 parts of water-absorbing resin (5) that had been produced in Example 1, 0.1 part of ethylene glycoldiglycidylether, five parts of water, and one part of isopropyl alcohol were added. Thereafter, a mixture produced thereby was treated, in a dryer, with heat at 100°C for 30 minutes. As a result, water-absorbing resin (9) was produced.

Water-absorbing resin (9) that had been produced in the manner described above was analyzed, and results of the analysis are shown in Table 1.

(Example 10)

To 100 parts of water-absorbing resin (2) that had been produced in Example 2, one part of glycerin, six parts of water, and one part of acetone were added. Thereafter, the mixture was (i) putted into a blender whose jacket had been heated to 230°C by a heating medium, (ii) mixed, and (ii) treated with heat. As a result, water-absorbing resin (10) was produced.

Water-absorbing resin (10) that had been produced

in the manner described above was analyzed, and results of the analysis are shown in Table 1.

(Example 11)

To 100 parts of water-absorbing resin (3) that had been produced in Example 3, a solution composed of (i) 0.1 parts of ethyleneglycidylether, 3 parts of water, and 6 parts of methanol were added. A mixture produced thereby was treated, in a dryer, with heat at 130°C for one hour. As a result, water-absorbing resin (12) was produced.

Water-absorbing resin (11) that had been produced in the manner described above was analyzed, and results of the analysis are shown in Table 1.

(Example 12)

To 100 parts of water-absorbing resin (4) that had been produced in Example 4, 10 parts of aqueous solution composed of (i) one part of aluminium sulfate, (ii) one part of glycerin, and (iii) 8 parts of water were added. A mixture produced thereby was treated with heat at 200°C for 30 minutes. As a result, water-absorbing resin (12) was produced.

Water-absorbing resin (12) that had been produced in the manner described above was analyzed, and results of the analysis are shown in Table 1.

(Comparative Example 1)

The same operations as in Example 7 were performed, except that 0.273 g of cross-linking agent (B) (0.004 mol%)

with respect to monomer (A)) was used. As a result, comparative water-absorbing resin (1) was produced.

Comparative water-absorbing resin (1) that had been produced in the manner described above was analyzed, and results of the analysis are shown in Table 1.

(Comparative Example 2)

The same operations as in Example 7 were performed, except that water-soluble chain transfer agent (C) sodium hypophosfite1hydrate was not added. As a result, comparative water-absorbing resin (2) was produced.

Comparative water-absorbing resin (2) that had been produced in the manner described above was analyzed, and results of the analysis are shown in Table 1.

(Comparative Example 3)

The same operations as in Example 1 were performed, except that (i) water-soluble chain transfer agent (C) sodium hypophosfite1hydrate was not added and (ii) 3.41 g of cross-linking agent (B) (0.05 mol% with respect to monomer (A)) was used. As a result, comparative water-absorbing resin (3) was produced.

Comparative water-absorbing resin (3) that had been produced in the manner described above was analyzed, and results of the analysis are shown in Table 1.

(Comparative Example 4)

The same operations as in Example 1 were performed, except that (i) water-soluble chain transfer agent (C)

sodium hypophosfite1hydrate was not added and (ii) cross-linking agent (B) was replaced by 17.73 g of N,N'-methylenebisacrylamide (0.5 mol% with respect to monomer (A)). As a result, comparative water-absorbing resin (4) was produced.

Comparative water-absorbing resin (4) that had been produced in the manner described above was analyzed, and results of the analysis are shown in Table 1.

(Comparative Example 5)

The same operations as in Example 7 were performed, except that cross-linking agent (B) was not added. As a result, comparative water-absorbing resin (5) was produced.

Comparative water-absorbing resin (5) that had been produced in the manner described above was analyzed, and results of the analysis are shown in Table 1.

(Comparative Examples 6 to 8)

The same operations as in Example 9 were performed using comparative water-absorbing resins (1) to (3) that had been produced in Comparative Examples 1 to 3, respectively. As a result, comparative water-absorbing resins (6) to (8) were produced.

Comparative water-absorbing resins (6) to (8) that had been produced in the manner described above were analyzed, and results of the analysis are shown in Table 1.

4. Brief description of drawings

Figure 1 illustrates a test equipment for testing the liquid permeability of a swelled gel.

- 1 plate
- 2 swelled gel
- 3 paper towel
- 4 test vessel made of acrylic resin
- 5 inlet opening

Applicant NIPPON SHOKUBAI KAGAKU KOGYO Co., Ltd.

Table 1

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MBAA : N,N'-methylenebisacrylamide

TMPT : trimethylolpropanetriacrylate

PEGDA : polyethyleneglycoldiacrylate (n=8 or 14)

SHP: sodium hypophosfite1hydrate

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本発明は耐久性の優れた吸水性的脂の製造方法

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耐久性の優れた吸水性樹脂の製造方法 の発明の名称

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	明 者 是砂 欣也 大阪府吹田市西湖旅町 5番8号	明者,是一时,在一大一个一个一个一个一个一个一个一个一个一个一个一个一个一个一个一个一个一个一	明 者 長 砂	明 者 民 砂 庆 也 大阪府牧田市西海旅町 5 眷 8 号往中央研究所内留 表 廣 濱 大阪府牧田市西海旅町 5 犛 8 号往中央研究所内	明 者 民 砂	明 者 長 砂	明 者 長 砂	明 者 長 砂 氏 也 大阪府牧田市西海旅町 5 番 8 号 社中央研究所内 社中央研究所内 社中央研究所内 明 者 本 村 和 正 大阪府牧田市西海旅町 5 备 8 号 財 者 本 村 和 正 大阪府牧田市西海旅町 5 备 8 号 財 者 本 村 市 土 社中央研究所内 明 者 下 村 忠 生 大阪府牧田市西海旅町 5 番 8 号 現 中央研究所内 社中央研究所内 社中央研究所内	明 者 長 砂 氏 也 大阪府牧田市西海旅町 5 番 8 号 2 全 金 大阪府牧田市西海旅町 5 番 8 号 3 本 村 本 大阪府牧田市西海旅町 5 番 8 号 4 中央研究所内 2 大阪府牧田市西海旅町 5 番 8 号 3 本 村 市 大阪府牧田市西海旅町 5 番 8 号 4 市 市 大阪府牧田市西海旅町 5 番 8 号 4 市 市 大阪府牧田市西海旅町 5 番 8 号 4 市 市 大阪府牧田市西海北町 5 番 8 号 5 大阪府大阪市中央区高盟橋 4 丁目 市 大阪府大阪市中央区高盟橋 4 丁目	明 者 長 砂

数ち母戦役 (V) に払つたり、02~1 ホゲ%かめ る資水項1または2記数の製造方法。 1. 水函佐エテレン位不飽哲単貴体(A) および 京中町弁(Y) に対し、政政政(B) 0・005~6 耐久性の優れた吸水体振路の製造方法 2、特許研究の範囲 1. 発用の名称

ホルなおよび大衛位通道な影響 (C) 0.001~ しホッスかかなすら3の記載ス~包竹鑑賞の授与 とする群久住の仮れた吸水住街間 (D) の製造方 国体(V) 水油液化、水溶液固合させることを特徴

ト反びしうも数米在珠母及(E) で母信させること を特徴とする耐久性の優れた吸水性研鑽(f) の繋 2. 大部和スチワン和下部哲學即存(A) および 四年(Y) 大路流布、大路波線由中台广部介風水坑 芸服(a) の数固済部を吸水和逆暦(a) 中の西原権 数中華化(Y) になし、級研及(B) 0.006~6 モルスセンジ大部行盗政存職器(C) 0·001~ しゃっとからせしゅ 3 0 町町 2 ~ 気竹道(反) 5 年

3. 木色体温度移動剤(6) が次田は設備である 4. 政益証(8)の仮用質が火格的イチフン位下 請求項しまたは2記載の超過方法。

5. 长面有 N P D N 和 K 包包 B 取 存 (V) 长 咨 资 の態気か3の国際某へ西台道路である証状型1分 たは 3 記載の製造方法。

6. 水路はエチレン柱不動的単盤は(A) がアク またはアンホノウム量も少なくとも50種関名会 リル酸およびアクリル酸アルカリ金属塩および/ **んでなる調水項1または2記数の間当方法。** 1. 大路供メチワン供不然的基面体(V) 大路浜 も回転資料値を指する反応器内で、水路液理会な せることを特徴とする請求項1または2記数の製 8. 回転債体買を有する反応容器が債数の回転 9. 複数の回旋撹拌館を有する反応容器が双稿 数存成を付してなる数を扱って記録の製造方法。 国ニーダーにある請求項8に記載の製造方法。

既宜姓(8) の京田戦が大治なイナンン行ド ■午(A) に対して0.04~0.4モルK である讃水原のに記載の製造方法。

11. 欧米和亚施(D) 1 0 0 阿爾郎になった、米 0.質問題の存在下、ガルボキシル沿に反応しつる 0~2の異層形および数水体有機溶剤(0) 0~2 8 個以上の首組括を有する化合物 (E-1) 及び/ま

たは多倍食薬塩 (E-2) からなる耳から過ばれた少 なくとも 1 語の寛永位発送 (E) もの・0 0 6 ~ 5.質異態を読むし、40~250℃で凹熱するに

3. 発数の詳細な説明 (超無上の利用分野)

> 13. 服水佐羅は刻(5) か化合物(6-1) である語 ヒよりなる請求項3記載の取簿方法。 校項11記載の製造方法。

13. 化合物(E-1) が多値アルコール化合物、多 陌グワウジガメーチ 5 行会後、 4 廃斗 4 ナソリソ 化合物及び多億アミン化合物からなる群から過ば こと 1 独または 2 値以上のものである研状項 1 2 己数の監論方法。

ているため、生殖用品、紙オムッなどの衒生材料

かした、戯聞巻、は代用の破水艦とした。好に その打動な吸吸植物品の女 なとして行く祭用さき

かから吸水在形路に安倍に防倒に関当たき安全 住に優れているだけでなく、優れた吸水筋を持っ

方法に関するものである。

14. 米が吸水布金融(0) 100風戦略に対した 0. 5~10個個四の最大用いられてなる間状況

特に勝潤ゲルの耐久性、経路安定性が上記特性に しぐしながの、改米和逆艦の圧割によったは、

【和訳の故稿】

数類を適由なな低として患さるッなどに困いた姿 51. 大人必要となってくる。 53.4 だ、 将米の段米机 4、展外数段した段を私位館の研覧がかが結群的 に次介した少なも近にしたり、また親国牧師など の央原因の使用で劣化し分解を起こすことがあっ 近年、白風の数10倍から数100倍の水羊吸 良する吸水柏波部が尾鳴され、概ちなつ、中間形 ナプキンギの街材分野をはじめとして農林銭分野 この様な風水粧服路としては、倒えばポリアク 7. 7. 数据分中的钻路模块(邻配配55-8430 士木分野等に幅広く用いられている。

いれまかに包のれた?も段水和逆器の矫蹈ケグ の発化、分解の形式が沿れていたは吸水体短幅に合 数杯道元件紙魚道やラジカル道板禁止層などもむ の包木分杯物(存公路49-43395年)、数 4 年)、戦災一アクリロニトリルグラント組合体

3 4 8 号)などがある。しかしながら、これらの 女のよる方法(存取因63-118375年、 特 随股63-162867号)、数化耐电台存させ **る方法(特別組63-153060年)、無質含** 有道元机を合有させる方法 (存属配の3-272 **力液はいずれも吸水粒光路に光穴形計のための斑** 囚禁を囚える方法であり、他の恐怕政を加えると いうことは、それの風水和記憶が落生ななのどに 位-アクリル数エスナル共団合体の中和物(特別 これの欧米柏伊脂に図まれる存在としては、米 有液体に抜した蚤の角と吸収部部や優れた吸水道 質、木佐液体も白んだ路なかの液を吸い上げるた B 2 1 - 1 2 5 4 6 8 号)、啓観ピニルーアクリ ル設エステル共国合体の製作物(特配路52-1 はアクリルアミド共国合体の団木分解物(特公田 53-15.959号) またはこれらの果婚体がど 4689年)、アクリロニトリル共国合体もしく

が置られている。

便用されることを与えると、安全徒の国から必ず

めの現れた風引力などが求められる。

に 国 智 少 の 少 な 2 吸 水 有 被 間 以 段 水 倍 申 6 信 下 し

てしまうのが実信であった。 そこでは私が見る多く用いて母が竹形配の泉谷

しも呼ばしてものたはながらた。

また吸水性樹脂の製造時に盗奴移動剤を使用する ことにより収米倍率を向上させる技術が開示され しかした、この姿な欧大松蛇窟におこと球盗艦 **にとは公凶であるが、策謀地の噂が扱えればそれ** の配合量を増加すればするほど耐久性が向上する なな数大衛母が属下するという監察点があった。 分な耐久住を持たせるには、高原国のため吸水田 く、且つ群女性に優れた吸水体財職は得られてい 的資を貼むることで、ゲル協議を確め難久存を向 上させる方法ももろが、これらの吸水性は脳は光 このように既任、安全保に保む・政水部部が掲 母が南独に何くなっとしまりのが米証ともった。

Cいるmm (USP4698404) . しかしこの 集合、大や共国政治大に対する政大部邸は国かに 向上するが人家に対しては吸水倍率の増加は殆ど あめられていない. また、上記の耐久性に加えて、オムツなどに組 **か込んだ夜に夢路ケアのヘトッキを過渡有の舟下** などが四国になることがあった。政大位部録には

覧って本発明の目的は耐久性の優れた吸水柱が節 本発明は上記項状に値みなされたものである。

品に盆を込んた路に共口のストシャのため過ぎ款

よ。)が存在し、これの水性筋分のため、数水袋 の閉鎖ケグかストシょうり、キオッなどの免疫物 ナニとかめる。 大国治少母は一般に吸火治事と応

の価因関係にあり、水凹部分原域のためには吸水

が向下し、足れな眠が存出かれた路にホフキ店に

在液態の成成砂点を上げなければならず、その角

団ク群ギムシトの取り観も少なへ、 グブのストシ

[乾眠が然沢しょうとする眞田] 大浴位となる間分(以下、これも大円箔分と呼

ななに人民になしたも治で吸水商品を示し、使い 大路路の街の田的女、中国女話大に対しても非 **都てオムツに使用した指に張れた耐久性を示し、** の観覧が訳を超供することにある。

> 中国が少なく国政体に優れた吸水性迷路の駅遊方 浴を取供することにある。

本能够的方式,上仍因因为解决于人人统即被对 (課題を解決するための年段及び作用) した結束、 (1) 存成明の配位型(8) と火衛特当益移動型 とにより、欧水循母を高く保ちつつ、耐久性が向 **寸し、米旦路分の分十数の角下に弁いかがのスト** シ 4 昭 4 過 液 供 6 改 啓 8 れ た 回 好 な 吸 木 体 遊 脂 (c) やむせつに犬箔紅メチァン紅木包竹単貫体 (4) 火部資を存取の運用配配で水路改算合するの (D) が出稿なよく等られること、

させることにより、町に扱れた吸水物体の改物数 、ケガのストシャ節や道路和が巨大し、欧米存在 右並語 (b) の数因消吸や成米特殊確定 (E) も秘護 (1) さのに上記(1)の取過が扱い序のれた殴が 哦やボン、 篤と段大節母も保むしひ、より既久积 に優れた吸水位部間(F) が符られること、 を見い出し本釈服を光改するに関った。

大価板イチレン4件/60台母者は (A) および以 4点 **もなする30値数%~数管鎖膜の損母循体(4)水** 20 液木合物 単の音形式、木筋液面合きせることを 降扱とする耐久性の優れた吸水性麒麟 (D) の製造 井(Y) になり、80歳屋(B) 0・006~5キル% 长部存当数が存む (c) 0 · 0 0 1 ~ 1 キガ%を 光光

(俗位メチレン位下位台中間体(4) および以が氏 存(V) になし、単独型(B) 0・005~5モグ% **米苗供道数移動型(C) 0.001~1 モル%を** ↑ 位する30 気動化~包包浴(M)の設存 国体(N) 水 3.後冬4年代の存在形、火焰波型のかれて年た日 大有热阻 (1) 多数阻消参先员长在进程中的包括指 1.反応しかる数米有磁管器(E) か根據かせやいか と特徴とする群久住の遅れた吸水位出頭(E)の敷

本税服をからに詳しく説明する。 に関すものである。

本処既に無いる大部額メチァン将不結的単曲体

町ち、本兜明は、

でき、これらの群から過ばれる少なくとも1種の るものであり、例えばアクリルは、メタクリルな トレイン数、ファール数、クロトン数、イクコ アクリルアミドー2-メチルプロパンスルホン製 アクリルアミド、メタクリルアミド、2-ヒドロ チツドチガ (メタ) アクロフート・メヤキツだり **Hヂケングリコール(メタ)アクリレート・N・K−** シメナルアミノムチル (メタ) アクリレート・N. トンスチルアミノブロビル (メタ) アクリレート . K.K-シエチルアミノブロビル (メタ) アクリル アミド、及びもれらの四数値などを挙げることが (4) (以下、母町荘(4))とした兵和馬伯を仕上 ン型、カイダムンカンペグボン製、ロー(メゲ) 21(メタ)アクリロイルドケンスアポン段、 2 - (メタ) アクリロイルプロパンスルホン殿、 及びそれらのアルカリ会員道、アンモニウム値、

クリルアミド、 (ポリ) メチレングリコールジ

行中替とした。 安えばN・N・ -メチレンドスプ

(メシ) アクンフート. (まい) どロパフングン

コールジ (メタ) アクリレート・グリセリントリ (メタ) アクリレート、グリセリンアクリレート メタシコワート、(メタ)とクシケ類が自分配前 . トリメチロールプロパントリ (メタ) アクリン **ート、トリアリルアミン、トリアリルシアヌレー** ト、トリナンガインクアヌワート、トリアリガホ スフェート部を挙げることができる。また反応位 首柄指を掻するものとしては、 吸えば、 単世体

なかでも上記の単国体(4)の内、符られる風水 性患脂の性質やコストの点からアクリル酸を共成 分として用いることが好ましく、もの既はアクリ

反応性官院首を対する保護剤(B)を特定量用いる

また本地野において耐久性の優れた吸水位更語 を得るには分子内に 2 個以上の関合性不飽和為や

5回車%以上とすることである。

7.数及びそのアルカッ金属指および/またはアン ホークム猫の位佐母は単国体 (4)の内で50種間 24.以上とすることが呼ばしく、見に呼ばしくなっ

16日平3-179008 (4)

にてが必須である。これの既信政(B) として聞え 17、分子内に2億以上の配合性不均的指を対す5

温度移動類 (c) を用いない低は固一条件で行う場 (4) がカルボキンル路を有する場合には、エチレ ングラコード、ジオテレングリコール、トリオテ

、現でなましくは4~10年モルである。具存色 **台の保証料の依旧観に比べた、2~100倍モル** には、脱硫烷(B) の食用質は単氮弁(V) に対した の食圧割がり、005キグス状態と共命のたぐ及 大有逆語の皮大節母は強いものが降られるが、野 久存に劣り回う米町路分が多へその分子買も殆い **ため ゲアの ヘトッキ 磨や 過減 存 に 光っ たもの て な** る。またロチル%を超えて多く使用すると吸水笛 母が学校に向いものとなってしまり、また既位題 の使用量は後述する水油性遠債(移動剤(C)の使用 異にもよるが、好ましくはひ、02~1モル%で もり、風作がまつくなり、0 4~0、 4 ボグなか あり、型により好ましくは0.08~0.2モル 0. 005~5ホルメナもる。これら原位形(8) ワングリロール、共リイヤフングリロール、プロ パワングシロール、ジンロガレングシロール・ボ シンロのフングシューダ、グッセシン体の砂角と ダローブ胚:(ゼン) イヤフングコロードシグコ シシアメーチガ、グリカロールボリグリシシチメ しから 降の 安値 グリッジ かた合き ちょ イチレンジ ア **ミン、ボンドチフンイミン群の安局グミン・ホの** 当、少何キキセンリン穴の位。スロメボキツ穴の **此心分子内に蛋合在不然的指人反応在百能器を合** むな 待しらのとしてはグリシジル (メタ) アクリ レート、N - メチローグ (メタ) アクジグタドド

って保備を形成させる方法も併用してもよい。こ の事なが浴として、セグロース、微妙、ポリピー また上記與情報(8)の他に、グラフト整合によ **カア アコール 等の観水住地分子の存在下に単数体** * 1.80.

のなかでは、分子内に2個以上の国合性不飽和基 5.有する化合物を用いることが得られる吸水性斑

降を曳げすることが出来る。これのの保存院(3)

な、今 阻 インシアキート、 砂 固 倒 随 西 夢 夕 め り、

3.85とする吸水価単の吸水柱を、守るために水道柱

これの保護型(B)の本部既に於ける使用概は 脂の耐久なや吸水体性の点から特に好ましい。

本発明は特定因の水道性遊り移動剤(C)を用い 5ことが必須である。

住途貞移動稿 (c) を選び、且つ過常の製造方法に 光気質として近台を行うことで切めて、 耐久性が 非常に優れ、生職食物水のみならず人尿に対して も話い収水価単を示し、また水可商分の分子量が **肩いたむ、ケアのストシキ筋や脳液布の肉下なか** の趣思智が少ない等と合う、優れた吸水住根脂が トロデガス・3ン。 大式によった。 本発色の和大量は新加いされる。体配面の水面 **元くて2~100億キルと参属の経済性を設用し**

米苑県に食用する水磁石磁盘(存物型 (c) とした は、水または火焰なスチレン体不動物単位体に溶 **弁するものであれば特に虹吸されず、チオール規** 次部体数値数などを挙げることができ、具体的に ナオール観視、2級アルコール紙、アミン暦、

ンプロパノール、次単項数ナトリウム、縁載、お よびそれらの複数が原用され、これの群かの過ば れる1種または2種以上が用いられるが、その効 原から次亜幼似ナトリウムなどの次型は砂塩や用 ロメアなントトケノーで、メアなノトゾロバノー チキッンゴ鉄、3ーメアケブトプロガキン袋、人 み、ドナシガメガセブナン、チャグリコール酸、 いることが好ましい。

とちめる。この食用同なり、001カケ光米遊り またしもアダル超えたぞく配用すると犬凹箔分か 长语在强重移物数(c) の倒用無以水浴在遊览符 智慧の協邸や原用政、専輯な (v) 水苗液の設成に 6 よるが、単質な(1) に対して 0・0 0 1~1 f イギンもり、 子まつくなり、 006~0 コホブ は、本党の政策を(8)の使用的では政権の対 4480円、 単久供がかえって倒下するので呼ましく 拓へ段 水節 中が角へなっ すぎん なまつくない

・しては30回草な~気竹部房、よりがましくは3 本常用に用いられる母割石 (Y) 水沼奈の設成と

5 母母女~名谷後庭の高田さめも、3 0 毎年を 朱森の道氏では単位反応容領当りの先盤性が下が 9、また乾燥工程にも時間を乗し、生産性が低下 缶では生傷性向上のこめぬ名為漢付近で重合を行 の間が殴られ、そのため配久位にだしい敬水在世 によった四川飫拉区乃か与陸かれ、桜在柱(8)の して工業的観点から好ましくない。好来の国合方 **うと自己経位などの不暇な反応が配こり吸水部等** 暫しが降られなかった。しかし女発用の方法によ たば、水道在過点移動剤 (C) の倒用書を過ぶこと 使用量が始やせるので耐久性に優れた路根水倍率 の良米有史語を塩釜屑に出層有よく雙鎖すること か用下するため、自合等に使用できる発摘剤(B) 4486.

また必要に応じて単数な (A) 大部派に基格部の 安用してもよい。この神な草枯燥としては、 倒え おおり アノ みどり アン・ ポンアクシ ルアミド・ メチクカグロース、たドロチツバチグカグロース 昨先なげることができる。

困ち吸れた数水石製脂も降らが沿として水油液配 合が採用される。 木谷液固合を行う方法として は、例えば型やの中で行う柱型虫台(特公昭48 - 42466)、スプトロンスソードも図むする 方法(韓魏昭58-49~14)、台水グル状版 合体を指分化できるような質な別数を有するニー 体を弱を得る 斑合が流としては、公路の 型合技術 たよる舞台などの中で、依然国や虫戯性やコスト 雑状質白。 紫外質や電子類などの語数メキルギー ダーなどの中で図合する方法(特別配57~34 ,例えば水路後属合、近格局湯既合、沈殿既合、

資移動所 (C) による均一な過貨移動反応や保債剤 (8) によるねーな厳協反応を維行させ、より体験 の向れた政大和安徽も称やためには、関心死が古 一に猿虫されることが呼ましい。そのためには固 **台ゲアジー弁となる医台沿ではなく、風合の配形** から故しまたの虫萃詰あるさな一部た、好ましく は金藤間で反応系の舞台ゲルが抜拝され聞合祭の この遊な水路液阻合が行われる場合、水部住道

101)などが既示できる。

台反応は国際協作関を有する反応容益内で行われ ることが汗ましい。回転放存就を対する反応容益 い虫皮する型合ゲルを固信貨件翼によって質断力 用いると、質色ゲルを踏かく組分化し組合の金属 因にわたりなーに気体ができ間合然のねーな存状 森林力が大きいものか好ましく、 包合の選行に停 破、一種の部出版、双発型に一ダーおよび川館に - ゲーなどがおげられる。また以鹿型ニーダーや を行えるので、より住能の優れた吸水住街籠が得 均一な縁去がなられることが呼ましく、よって田 かつとは、むら髪をすな?が、風のゲケになせる と与える反応値なども倒示でき、また奴件力を大 4 ヘナもためにな回帰道が就は複数ためもことが よりがましい。 反巧位として 弁包 人式一名の 強数 られるので許ましい。

14、過数化水素、アン化合物よりなる群から近ば

れる1組または2種以上が呼ましい。

17 台台:もの名、第二カリウム塔、過トンガン袋 国体が挙げられる。中でも除られた吸水柱出版の 住経面や分解生成物の安全性等の点から、過程数

キャメド降のハイドロ パーよホケイド: 2,2'ープ ゲアス(2-アミジノゲロバン)川南政治学のアン

排間平3-179008 (B)

段名別の益令、協巧堂令守田つトファックス成民 右盤とした思いたる扱い。 無いのれる過形盤とし

またりシカル風台配格型が設に位うジャル組合

リケム路の間隔裂(水軒)類:チギ鶏繋ナトリケ 4 年のチャ民製品:街「チェン製品:風製彫一屋 、攻撃隊一様命の句呵茲:4-アスコルアン設等 の有額協咒器:アニリン、モノエタノールアミン

ては、例えば難説粒ナトリクム、難残数水煮ナト

れず、例えば過級数カリウム、過級数アンモニク 19 20 企本表立 ム、過級数ナトリウム等の過級設置 デモープチル 女路毘になた、女治疫威合に食用されるシジケ 7.氏白配着近としては火部なりもれば体に怠困か ハイドロ パーキキサイド、 クメンハイドロバーキ

ラシカル属台居治剤の使用度は広い範囲とする 穴がましくれる。 0.1~0、 5.4.7%の配面かめ 001~24万%の原田でするのがなれてく、区 にんかたきるが、過化、母解符 (A) にだして O.

母のアミン型が挙げられる。

条数で、80℃年中では乾燥に関係がかかり出産なのでは一次である。 2度42で、よび42で、また230℃半十年年の水 姓もれた人ケトまたなドシムドートをも正り 用い、重合後の含水グルを80で以上で、より肝 ましくは80~230ちで気気することが好ましました。 食品 る民種方法が挙げられる。これらの乾燥方法を な影響の劣化が配こる場合があるので往殿を取す

また以上の様に群合、乾燥して終られた吸水体 班指は、必要により的なおよびまたは分徴して欲

なのに本発明は、哲記製造方法によって符られ 5. 良水供飯間 (12) の数固消瘡や存配の数水供成塩 E(E) で紙織のせた数米供金額(B) の豊裕方法を も遺供する。本発彫の敷配製造方法によって得ら **筑状の晩水有無路に孔くれ着しい晩米な有の牧物 兵牧園近夢も経緯する他の呪水私豊穏(0) に共** 人。女乡に耐久住に優れ吸水体住に優れたものに 谷典本示し、教題近都を成績した母本校建語(6) 九九夏木住田曜(8) は、李明近日の平日 mene.

々。この食用質が0、001キル%米減では減合 協語のため、概合疑的監観を確加させると自己祭 **らら、食用できる民谷近戯も殴られてきた,いたか** 超額中原連越間が長くなり、また既存モノマーも **ゆくなりむくびましくない。また紅米の国合力沿** ナス、私存モノヤー(自然や記録部院、組合保証の ほなどの不取な反応が配こり吸水価等が低下する 本兒明の方法によれば、かかる欠点は改善され **ふたも・しかつるホテスイン多へ展用したも、時** 旨無に見合った効果か少ないばかりか、難合反応 なその組合品により乾燥と配合を回路に行っても 良いし、 組合徒の合水母によっては得られた合水 ケルを更に吃益することによって吸水位が踏とし て使用してもよい。乾燥方法としては公知の乾燥 力浴を用いることができ、鬼人は有臓器剤中たの 医铬粒固に指信させたも施供館の政水在建設が結 本院配で等られる段木体が設は有機設で配合を の慇懃が困難となったつまいのた呼ばしくない。 共海政水による方法、協製道風炉、被田院協設、

マイクロ液乾燥器、瘀外線乾燥器、胚低温度に加

本発明に於て単型体(A) 水溶液を固合させ吸水

本発明に於て使用される摂水性保護剤(E) とし ト は ガ ゲ ポ キ シ ケ 猫 と 反 巧 し う も 宜 信 組 も 1 分 子 国会員道(E-3) である。別人ば最大性出語(0) か としてはメチンングリコール、シスチレングリコ した、トリステレングリコール、チトラスチレン ソシオール、1,5-イソタンジオール、1,6-くキサ 中に2個以上有する化合物(E-1)及び/または多 グリコール、ポリメヤワングリコール・1,4-ブク カタボキンが組をなする場合には、代合物 (E-1)

ングリコール、グリセリン、ボリグリセリン、ト ルコール化合物:エチレングリコールジグリシジ **ホオーチガ、ポリメチワングリロールジグリンジ** 5、シグシカローケボングシックドードで、 4 **リグコセロー ひおしグリシジルドーチル、プロド** ワングリコールジグリシジスメーナル、ポリプロ ンジオール、キオペンチルグリコール、プロピン ルメーテル、グリセロールボリグニシジルメーチ ングハヤーグ、ボンハイグレテリーグ降の少国ゲ リメチロールプロパン、ベンタエリスリトール、

パケキャナンリン年の中庭キキナンリン介の甘; 2 協以上を用いることが好ましく、中でも化合物 (E) として、化合物(E-1) と多面金属塩(E-1) を 物などが挙げられる。これらの群より1種または (8-1) や必従に無いもにわがおましく、 参に夕雨 アルコール粒、多角グリシジル代合物類、多角ア ミン盤も最大性保証制(E) として用いることが数 国政権を果の点から呼ましい。また観火有除権包 木の筍の多個アジリジン代白物、多価インシアキ ート化合物などが挙げられ、多価金属以(E-1) と しては亜鉛、カルシウム、マグキシウム、アルミ コウム、狭、シルコニウム等の大数化物及び路代 ハフングシローチングニック ケメード 子本の 学庭 シスチレントリアミン、トリスチレンチトラミン 、よりイチワンよいソ谷の中国アミンの白色: 1.2-ドテフンガスキキャンコン・ガジムンプロス よアクロロドドリンなどのハロメボキシ代合物: 本院県に吹て食用された数水体保証的(E)の食 伊用して用いて混合位を向上させても揺りない。 グラツジケメーナが行む物:イヤフンジアミン、

> は上記、製造方法によって得られた吸水柱出路 (D) 100日放送に対して、0、005~6日時 8、 年ましくなり、 0.1~1 四親間の範囲の整合 ためり、この配田内の関わめた戸路存在に抜さた 在成设型(E) の風から四風節を超える場合、不穏 浴となるばかりか、米段氏の以木供原ඛ点(E)が 部のれた吸火な光照(E) に残なする凹筋なかめる 上に、道圧な果構効果を造成する上で過剰重とな りほられる段大な世間(5)の段大部中は小さくな **戦団部資益十の改大哲学語(5)がほのれる。 観犬** ではましくない。また0.005回転等米液の少 ない値か行を配配の悠保が行のれにへい。

本的時に於し、吸水位世間(D) と見水位原構成 (E) とを混合する塔、水及び/または貫水柱有益 遊覧(C) 今田いたもよい。

また数水位在資格型(0) として倒えば、メケノ 本既既にだた、その創産国に改そ存産婦(0) 1 00回間思想が20時間整理性、学者しくれの。 5~10風敏部の配面の型合にある。

ール、 エタノール、 n - ブロパノール、 180-

カトン、メチグトチロケトン、メヤプンチロケト ン等のケトン類:シキキセン、ケトサヒドロング ン体のエーサル数:H, H-シメチルホルムアミド枠 のアミド雄:ひメチアスクホキツド等のストホキ シド酸もおげることがため、その使用調な吸水体 プロパノール、ローブタノール、180-ブタノ 一万、 キーブケノート等の角数アルコール型:ア 安章(5) 100年間野に対した0~20年間部、 P# しくは 0 ~ 8 超最間の過回の配合である。

(二) 呪水仏世鑑(0) に収水仏球構成(6) および 必要により、水(水瓶気)及び/または銀水位荷 本路跟冗仗人,段米位进路 [0] 本线水标咏位档 (6) と混合し、吸水位出脂の数固近像を現場する 【新姓 (c) の後の漢を受禁、あるいは終下後合す 方法としては、以下の姿なが沿が曳斥される。

光代 4 ななな。 (11) 良水供班服(10) 多四台不招供存货水供并贷 治烈中に分散・慰濁させ、根水佐果協利(E) およ び必要により、木及び/または豊水佐省植物剤

はその乳化液を吸水柱側路(0)の分散態路液中に 如冬用いて、予め破火在女優洛利に乳化、 節減さ この場合、親木佐殿協政(E) を移配の界圏活住 [6] 全值样下に弱加する方法。

(8) 吸水柱拼胎(0) 多水上髓水铁有磷溶剂(6) 設督する仏社が卒ました。 선산

を記こすのみならず、規水性保護制(E) の一節が 来反応となり得られた吸水性掛路(f) に残停する 恐れがあり好ましくない, 250℃を超える推奨 では、夏木谷田暦(D) の道域によっては慰光化が

4 0 10 米国では反応に時間がかかり生産性の低下 より好ましくは90~220℃の範囲である。

指類などにもよるが、40~250℃が好ましく

加熱処理過度は用いられる数水性保護的(E)

の调合溶剤中に分散させ、現水性與偏別(E) を添 などがなだのたる。 近年の十四

なお、数固統領した吸水体が語(5)や、数固以 協していない吸水住労脂(0) を必要により破砂治

自してもよい。

聞いる場合があるので注釈が必要である。

の智慧器や幻然部を用いることができる。 曳えば 的核烧谷,英数磨轮烧替、免液吃烧器、积外蜂吃 後野、誘耳臼熱に魚粉などである。又(m)の方 法人员大位出版(0) 元有物俗对中个负水化保证列 (E) を恐怕した後、角類治剤中でそのまま如熱し 以上の様にして、本既用の方法によって仰られ S吸水性的脂(D) と脱水性原体剂(E) および必要 により、水及び/または親水性有機溶剤(G) を選 のしてほられた過令もを智然処理するには、過れ 省四层学总体部,回信党体器、用格党体器、行力

本発明により得られる吸水性細踏は従来の方法 しゃら木町路分の分子間が育へ、 形数ゲルのベト では得られなかった、堪遇グルの耐久性に優れ、 中国食品水や物に入販に対しても組免水部中で、 「雑命の珍珠」

れた毛存的の皮が有形態が認られるは、疣骸反か シキや過渡性が大幅に改容された、安全な吸水性 (3) 用いる単語存物図哲説図沿への植り姿図か 食のさせても、自己保護など不暇な反応が移えら 出版である。 本発明の方法によればごの様な扱わ た風水保護器は、水浴位ユチレン位下配台単野は

(4) 本规码の吸水技法路は高い吸水倍毋は示す 6、 女路する大国宿公はその分子組が廃いため、 AS 可治分による研覧グルのペトッキや過数在の印下 国合が耳続て高い生態技で呪訟がたきる。

気体彫刻 (c) の存在下で水溶液風合を行うだけで 医原に繋道とき、町に年のれた段大有金箔 (0) の

(4) 冬体院園の梁信頼(8) 及び特託園の米裕供議

吸水通风中耐久住の信贷に向上された吸水性断距 (F) かほられる. かかる吸水性逆脂(D) および (F) は、安価に製造できば来にない優れた吸水体

数固近છも特定の現水柱際偏袒(E) で保証させ、

幼果を示し、更に優れた耐久性や吸水道度を示 (5) 牧岡近安を銀位することで、如米の殷水杭 独語の教園部首か節のれなかった現木特在の改造 などの題影響を及ぼさない。

自己領域などの不安な反応があれり強吸水苗中の (6) 従来、発存モノマー低級や誘導期間、団合 うのが第のたなかったが、 真色配着性固を詰やし たる角曳大母のものが節られ磨い投存もノヤー質 時間の協語のため組合配格が関係を指記されると、

箱ゲルの町久住が乏しかったが、本見明の方法で

な多くの凝核剤を用いても、 苑段水市中なため、

筋温ゲルが優れた耐久性を示す。

(1) 标来、路级水倍母の吸水佐道脂を得るため **に秘資監督の返のつト四位を行ったでたため、班**

段第などの分野に揺広く利用できる。 <u>製造す場によ7億5人店</u> この様に本発明の資水塩用面数と4~の間角が常

在を在しているため、適生材料、食品用、土木、

中の優れた段所を対するものである。 622

(1) 従来の吸水性妊娠では得られなかった、人

尿に対する難い吸水価値を示す。

-91-

東衛の1に対し、用いられる大衛抗道位移動制

(東柘田2)

関係が入を吹き込んで浴存数据を追い出した。

2 1 9 g (対単数体(A) 0.05モル%) とする

以外は阿強に行い、収水位出路(2)を得た。

(c) 次囲袋塾ナトリクム1大台物の側用駅を1・

11 00 06 大位忠昭 (5) 今回第5 好因),結成今節

1 我に示す。

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Ξ	_	8	۶

炎珠安に記載の数木柱災難の諸物 み取託されるもの 独は下記の試験方法によって過節した個を示す。 また、既は四四四を示す。

以大在出版 1.0 6 年 1 5 0 6 10 0 . 9 年 2 % (1) 生理女協木の吸水価中

し、やっくりとグネチックスターツーが結びし た。の時間後に疑問ゲルを金越で強迫し、十分に 水むりをした液の閉路ゲルの値もを望信し、以下 **西代ナトリクム状部浜中の入ったピーケーに姿疚** の女で吸水田印を甘出した。

役木在法語の田曹 羽臼ケアの日田 **6 大田田 (8/8) = -**

(2) 人鼠の吸水倍率

のサングリングした人民を用いる以外は民欲に行 (1) の生母女塩水に代えて、成人男子10人か い、人民での役木命母を望信した。

中に分数し、12時間後、諸氏で編造し、協設中 最大在登録0. 5 m ~ 1 0 0 0 m 1 の 数 / ポン 水 の固形分を倒にした次式に従った水可箔分を状め (3) 木可溶分

は近の質問(E) • 資液の固形分(X) 0.6(£) 水可溶分(%) = --

(4) 水可陷分の分子型

・中午朝が筑凶の路々のボリアクリル版ナトリク る今紋母につた、ゲグバーミメーツョングロシド グラフィーによって、(3) の方法でサンブリング した水圧筋分の分子数を状めた。

不超市、雄杖パルブ、風水概ちよび防水フィル (8) 陽函グルの耐久性

ムからなる市覧の子供用オムツ(位配72m)を 半分に切り、ポリマー2.5mを掲げパルプと吸 水斑の間に均一に収拾し、成人の人際 1 2 0 mlを 位えて37℃に校覧し、6時間、13時間、18 な問役によるシを聞いて中の規数ゲルの根子を献

大部僚を送入し、国際ガスを吹き込んで反応が近 5. 国 株 路 板 し た 。 し と た 2 本 の シ グ 4 国 沿 曲 4 回 成ななられ状に、ジャケットに36ちの過失や過 2 6を透過することにより概合を認路をせた。 典 色ピーク時に台水ゲル蛋合体は芯 2 anの 猫に苗分 穴されており、更に資料を扱け間合を配拾した 6 0 分後に倒るがし、グルを取り出した。 ほられた アットなもステンフス監及限型なっか型(パーグ **ひん 苔続 つなかの 食布配 寄屋 かつ ト遊 破野 ゲン 布** 8. ケゲア状菌合体の指数代徴を5.0 メッシュの曲 現上に氏げ、110℃の遊倒で50分割影覧的機 一)に関系しげ、この反応器に上記の単数は(4) ニウム2.62mと耐腐穀水格ナトックム0. した。この先位也をハンマー型的存品でおなし、 2 0 メッツィ倒越たゆゆでむけした20 メッツ 内容賞 104のシグト型防御を2本性するジ

英語聞」に於て、用いられる原理部 (B) もポリュエナングキンドド44年代代 コチレングリコールシアクリレート (n = 8) (東語型3) (3) を符た. 一数几次十。 (東路配4) 运运卷0000大杆型器 (1) 多体化。

1.97 ほ (対単版件の、9~4人名)とし、かつ 大治体滅損移動剤(c) 次面偽設ナトリウム1水粒 もの後用質をB・78m(対単質体(Y) O・4 や 5名)とする以外は同様に行い、収水住倒脂 (4) を紹介。

> 粧1因に応すべつに内図63 meのツャーフュ 级大位出籍 1. 0 g & 入れ、人联 1 0 ml を注ぎ形 留ゲル2を飾る。既選ゲル3の上に再編63mgの スーパータルグひも何か、 別に送し囚にボナけつ 12、 中央に収出した値状態を対する圧旋状のアク ファ 労働 観覚 製物 4 本質 4、1 多国海道に収録し た後、人尿 6 ≥1を住入口 6 より在 6、人尿が全て ポリケー中に敬収されるまたの邸題も望然し、 チ

(8) 閉窗からの追答和

ė,

作した。光代状態が対解は、O~A~×の3段階

な回れした。

×:明政ケルの形状や恐れ、ドロドロの流息状

となっている。

(6) もどり出

△:一部、形置ゲルの形状が凝れている。 〇:研凶ゲルの形状が保持されている。

の子供原オムツの不穏布の上に20斤りにした2

上記形面グルの耐久性試験に用いた18時間後

4 O g/co. の用力をこか置かなたベーバークオル

に戻ってきた妖の動を遊覧した。

(1) 研覧からのくてかず

3 cm * 2 3 cmのペーパークオルを10枚かぶせ、

この処水布逆路(4) も同味に評価し、結果や黙 (米質欠5) 我们所有。

N. ーメチレンピスアクリルアミド2、152 g 【灶戸園弁 (Y) 0.06 モルS)とつ、かし大浴 性道道移動剤(C) キチキリンゴ酸3.48m(丸 単数件(V) O・1 モル名) とナも以外は国役に行 状態の 1 におて、用いられる妖紋型 (B) な N。 14、 段米有遊腦 (2) 布容化。

この段大位法語(3) 今西後に好成し、枯眠や咸 故に示す。 (東橋左6)

037組織的大部隊43808、台上び東京教 (8) としてトリメチロールプロバントリアクリレ

(1) た欧大田母や肥低した彼の明覧ゲチのスト シチ(ドシム的)の中省のホト程成つた。ドシム

アクリル酸414m及びアクリル酸ナトリウム

にかもって、男郎ケアの当後有とする。

(地語記1)

41米粒物0.195g(对母数体(A)0.00

8 先 少劣)、 イギン女祖大610g 先用いた道威

37%、中哲母76%の単型体水遊遊を部た後、

×・明廷グラダスマシサ、中グダスダスプトで

○:一部、閉路からかんトック。

、 长部섞道貨移物路 (c) とした次用は設ナトリク

〇:研査ケルはかなりセラケクしたドゥイ語が

的の型的は、○~△~×の3弦器は留とした。

以協吏」になた、 圧?のれる / オンズ 位 木 の 町 2 X かし、 眠 2 の 4 や 騒 4 2 (B) か 弁 フ イ チ フ ソ を50mに発更し、単画体 (A) 水極液の過度を A

アレングリコールシアクリレート (n = 8) を 5 741-127946-1 (ne14) 17.0 かつ水溶体道質移動剤(c) 次頭袋設ナトリクム 1 1 ホケ名)とする辺冬江回鉄に行い、欧大紅並短 この吸水供供路(3) を周抜に評価し、結果を兼 **以通史1になた、無いのれる秘信院(B) ポリオ** 21.988 (対型相体0.2モル%)とし、 水台街の欧馬戴を 2. 44g(対単町体(A) O.

ト R (対単戯弁(V) 0・1 R F R) とし、タレ长 8 B g (対単盤体(A) O. O 2 モル%) とする以 節位道数移動数 (C) ケオリンゴ酸の使用質を O・ 4、回接に行い、吸水供無(e) 4年た。

この数大和安徽(6) を同事に評価し、結果を選 1 数化示す。

(東部別7)

を聞えた反応44時に仕込み、55℃で1時間損件 (4) 0・3 木ルギ)、大密な国際存職等(C) とり 40mのトクモロコン製品及び600mのイギ ン交換水を提供はと路索ガス吹き込み替と温度計 後、30七に各世した。この政忠大協議にアクリ **か取300m、出資型(B) としてN・N・−メサ** アンパスプクリルアミド 1・92 m(女母母弁 C チャリンゴ製 O ・9 4 m (近年間子(Y) O ・1 5 小 万 火) 今 治 解 4 4 4 、 資 展 3 3 火 、 中 包 母 O 火

この母親存を始後も3日でとして、因の関わば として過度酸ナトリウムロ・20g、4ーアスコ の母的存大面供の存む。

Ê
特間平3-179008

し、10分後に殆ど既扱した国合グルを取り出し 、更に実績会」と回復に乾燥が砕し吸水柱柱間 ケアン数 0、04mを替えて3時回、反称しなが 6日の仕しむた。 ゆられたむ ボゲル 次国合物に、 3 0 延鹿なの哲在ソーダ木苗液を3 8 8 6 12 人、

11 0 0 0 大在金额 (8) 先回被后辞母し、姑果予郑 中台母20%とし、例に残骸医1と囚事に乾燥砂

この段が位出路(1) を周仰に呼回し、結果や部

一数に示す。

(以路田田田)

なつ良米有逆暦(1) やはた・

我们所干。 (多型速域) 美殖房17部られた吸水位出版(5) 100部に エチレングリコールジグリシジルエーテルロ・1

ち、大石笆、インプログライドローテー四条組合 し、毎のれた選合物を乾燥数中に100℃、30 少智台総名道を行い、 吸火性逆暦 (3) を除た。 アクリル酸12mに脱イオン水22.2mも凹 人、反ここれや中台位として結束88%の水線の カリウム49. 5mおよび栄益型(8) としてN,

この後にして命のれた吸水位退船(3) の分が粘

N. ーメサレンピスアクリルアモドロ. 1m (丼 兴明年(V) 0.065七万%)、大遊信道数移息 巻(c) としたナキンソゴ語0・326m (紅母町 存(4) 0、15 4 4名) 4 歴代部台)、建筑 4 0

成为 第 1 数 7 形 上。 (東部四10)

17、グリカツン1路、大日路、アセトン1時外部 城路医2 7 年6 七六 8 大有 光理 (1) 100 四

人、ジャケットも発露で230℃に包括したプレ ングーに投入し、適合と加熱処理を行い吸水柱形 間 (10)を移た。

この母職存水出資を10mに保護し、む5mmの

2、中12年10%の中国存大部省中国独した。

ながら皆然が流下に促った後、別に過風物アント

0.2 まな恐怕し反合を作った。 概合は四風に反動

こウムロ、1gおよび蝦夷艦水橋ナトリウムロ。

この役にして係られた吸水性資路 (10)の分が枯

2 7 3 g (对母鼠体(V) 0 . 0 0 4 电万%) 化 ナる辺々は回義に行い、光気の火気逆燈(1) 6年 この扱にして慈られた兄奴母木和趙暦 (1) の分

秋福史3 か飾のれの段长和遊職 (3) 100部 市 **ユチレングリコールジグリシシルエーチル0.1** 日、大3四、メケノーで8酉からなる芭蕾節道を 議合した。 ゆられた凝合物を乾燥値中、130℃

张春郎1数に示す,

(米部別11)

你想我会想 1 数几形十。 (北数配2)

域路安1において、水油有道症移動型(C) 次期 谷数ナトックム 1 米色を糸斑 哲しない辺外 井岡嶽 に行い、比較吸水性問題(2)を得た。 この妻にして年もれた孔良の木和金類(1)の分 价档果先期一数几所广。

この後にして得られた吸水体出路(11)の分析結

张仓路1数亿所少。

(共協的12)

で1季節智能的語り与することにより吸水体影響 (11)

不留力。

秋福宏4か節のれた殷米和遊頭(4) 100部に 段優アルミニウム 1 部、グリセリン 1 部、木8部 からなる処理大治派10時を流合した。年のれた 街合物を200℃で30分回匈勢為現を行い吸水

の食用調売3.41.E (以早與存(4)0.05点 東路安」において、水油在道路移動型(c) 次形 境限ナトリウム 1 水粒物を遊びせず、吸吸粒(8) (共長四3)

この妻にして年もれた礼数政大和思照 (3) のか 价档果全篇一数に示す。 (3) 未得た。

この違にした年のれた政长和武隆 (11)の 4 花稿

在田路 (11) 化研心

最有難一致に添す。

(比较到1)

状活史!にないて、銃笞殺(3)の資紙町やの、

58)とする以外は国祭に行い、共党政大有党権

実施の1において、水浴伝道位移動列(C) 久田 資数ナトリウム1大智物を浴路せず、原稿型(B)

7 3 g (対単四体(A) O、5 モル%) とする以外 RN, N' - X + 1 ' Y X X X Y Y Y X T E F 1 T.

この様にして係られた比較数水柱田路(4)の分 **所结果を第1数に示す。**

な母後に行い、兄奴母水存出職(1) を得た。

(比較倒5)

英格回りにおいて、蘇佐敦(8)を設置しない以 吟は回答に行い、光数数米位置層 (2) 免除た。

この旅にして係られた比較吸水柱逆路(5)の分 所結果を第1数に示す。

(北京888~8)

比数例 1 ~ 3 で得られた比較の水枯ط脂 (1) ~ 3 を 4 な 2 2 (2) を それでれ、実施例 8 と同谋の保存を行い、(3) を それぞれ、実施例 8 と同谋の保存を行い、

比较吸水体整盟(6)~(8)を得た。

この姿にして年られた兄友及水紅斑路 (6) ~

(8) の少庁結婚や難し数に応す。

E 0 E 可溶分 超磁性 前 久 性 分子量 (ft) (ft) 金h/11-% x/x/x 11109 20 1210 93 日帝をインの日田の 3 5.2 -0000009 0 0000 09 ======= = * 225 氫 90.0 9.0 90.0 ## 149.5 BE 3355 证货化助料 \$117782 SIP SHP (モルエ) 回 0.00 - 8 9 数水柱形器 保 语 社 TMPT TMPT 比较吸水性 3.3 153 8888 20

-

2

2222

アロアルトリンプランプート(n=8、または14) アロアルサナトリウム(木色物

-80-

- 20-

7

11.1 図は形型ゲルの過激体は数器具を示したも 4. 図面の配用な説明

7-+4 9485.

2 … … 弱効グル

4……・アクリル開路製貨級器 3…… ペーパークオル

5 注入口

第一区

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